PATENT SPECIFICATION

(11) 1 575 819

(21) Application No. 47124/77 (22) Filed 11 Nov. 1977
(23) Complete Specification filed 8 May 1978
(44) Complete Specification published 1 Oct. 1980
(51) INT. CL.³ G01N 25/04 //
F25B 25/00
G01N 11/04
(52) Index at acceptance
G1D 24
G1S 1M 1T

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(19) THE PATENT OFFICE LONDON

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(54) SYSTEMS FOR TESTING MATERIALS AT TEMPERATURES DIFFERENT FROM AMBIENT TEMPERATURE

(71) We, STANHOPE-SETA LIMITED, a British company of Park Close, Englefield Green Egham Surrey do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates generally to systems for testing materials at temperatures which are different from normal ambient temperature and in particular to systems of this kind in which the material to be tested must be held at a closely controlled temperature or subjected to a controlled variation of temperature.

The invention provides a testing system for performing a test upon a sample of a material at a temperature other than ambient temperatures in which the temperature of a testing apparatus is arranged to be changed with respect to that of a reference body, which is itself arranged to be held at a temperature different from the ambient temperature, by a Peltier effect unit thermally interposed between said reference body and said device.

The invention may be used to produce cooling and/or heating of the material with respect to a reference body that may be a heat source or heat sink, as appropriate, provided by a body heated or cooled to a predetermined temperature in relation to the local ambient temperature.

A substantial advantage of the invention is that it enables close control of the tempearture or temperature variation of the tested material to be effected by means of control apparatus requiring only low voltages for its operation.

The invention will hereinafter be more particularly described with reference to an exemplary embodiment, specifically an apparatus intended for determination of the freezing point of a liquid freezing at a temperature substantially below the normal ambient temperature, but it is to be understood that this application is not limiting on the scope of the invention.

Reference will now be made to the accompanying drawings in which:—

Figure 1 is a schematic diagram illustrating the arrangement of a testing system embodying the invention;

Figure 2 is a sectional side view of a testing apparatus in the form of a cell adapted for viewing thermal phenomena in a transparent liquid.

Figure 3 is an end elevation of the cell shown in Figure 2; and

Figure 4 is a schematic diagram illustrating an apparatus including a cell as described in relation to Figures 2 and 3 and arranged for determining the freezing points of liquids; and

Figure 5 is a schematic diagram illustrating advantageous modifications of the apparatus of Figure 4.

Figure 1 illustrates a testing system 1 embodying the invention in its general form. A testing apparatus 2 is required to perform a test on a substance 3 which is to be maintained at a temperature which is different from that of a reference body 4. In accordance with the invention there is provided between the testing apparatus 2 and the reference body 4 a Peltier effect device 5 arranged to produce a required transfer of heat between the testing apparatus 2 and the reference body 4 when energised by electric current. Electric direct current is fed to the Peltier device 5 from a direct current source (not shown) by way of a control means 7. Control means 7 may be manually adjustable by means of controls *7a*, *7b*, which may respectively be a current varying means and an on/off or reversing switch. Alternatively or in addition control means 7 may be arranged to respond to a signal received from a

temperature-sensitive means 8 and/or to commands applied to the control means from a program device 9, so as to cause the temperature of the tested material to be maintained at a predetermined value or to vary in accordance with a predetermined program.

One particularly advantageous application of the invention is to a system for determining the temperature at which a visually determinable change occurs in a transparent liquid material. In a system of this kind it is convenient to make use of a testing apparatus consisting of an observation cell of the kind illustrated in Figures 2 and 3.

The cell 10 shown in Figures 2 and 3 comprises a metal block 11, usually most conveniently of aluminium.

In the block 11 is formed a well 12 extending from a position within the block to an upper surface 11*a* thereof. A sealing plug 13 is placed in the mouth of well 12 and is sealed to the walls of the well by appropriate means such as O-rings 13*a*. Plug 13 is longitudinally apertured to allow the passage therethrough of a thermometer 14, of which the temperature-sensitive portion 14*a* extends into the lower portion of the well 12, so as to permit the temperature of a liquid placed in the well to be determined.

To allow the introduction of liquids into the well 12 and their removal therefrom, two passages are provided in block 11. A first passage 15 extends from the bottom of well 12 to the lowermost surface 11*b* of block 11, where means 15*a* are provided for coupling an external conduit 15*b* to the passage 15. A second passage 17 extends from a position in the lateral wall of well 12 immediately below sealing plug 13 to a lateral surface 11*c* of block 11, or to some other surface if preferred. At the surface at which passage 17 emerges from block 11 it is provided with means 17*a* for coupling an external conduit 17*b* to the passage.

Aligned bores 18 19 in block 11 intersect well 12 and each contains a close-fitting plug, 20, 21 respectively, of transparent material, usually most conveniently a transparent plastics material. Plugs 20, 21 are sealed in the respective bores in block 11 by suitable adhesive or other known means. The inner and outer end faces of plugs 20, 21 are optically polished, usually though not necessarily to be plane-parallel, so as to permit convenient illumination and observation of the interior of well 12. It will usually be advantageous to place the temperature-sensitive portion 14*a* of thermometer 14 within the portion of well 12 which is visible through plugs 20, 21.

As illustrated, block 11 is a rectangular parallelepiped, but it may be more convenient in some cases to employ a rhomboidal, or otherwise non-rectangular block. In particular, it may be advantageous to arrange that bores 18, 19 are not perpendicular to the axis of well 12, but inclined thereto, so that the viewing angle may be advantageously inclined to the horizontal. It may also be advantageous to replace one of cylindrical plugs 20, 21 by a transparent body of more complex form to permit binocular observation of the interior of the well.

A testing system including a testing apparatus formed by a cell of the kind described above in relation to Figures 2 and 3 will now be described with reference to Figure 4. This figure schematically illustrates a testing system 40 for measuring the freezing point of a liquid freezing at a temperature below nominal ambient temperature (20°C). It will however be understood that a system of this kind may alternatively be used for determining the temperature at which some other visually observable phenomenon occurs in a liquid.

The testing system 40 of Figure 4 includes a reference body, in this case a heat sink, provided by the evaporator block 41 of a refrigerator system. This evaporator, which includes an internal cavity in which a refrigerant liquid is evaporated to produce cooling of the block, is coupled by way of conduits 42 to a compressor and expansion valve assembly 43 including a compressor operated by an electric motor 44 fed from an a.c. power supply 45 by way of a control means 46 responsive to the temperature of evaporator block 41, as sensed by a temperature sensor 47 applied to the evaporator block 41. Assembly 43 is also coupled to a condenser 43 for dissipating heat extracted from evaporator block 41. The arrangement described above operates in well known manner to hold the temperature of evaporator block 41 at a predetermined temperature below ambient, for example −15°C.

The system 40 also includes a testing apparatus consisting of an observation cell 10 of the kind described above in relation to Figures 2 and 3, which is required to be capable of being taken to a temperature substantially below that of reference body 41, for example, to −50°C. In accordance with the invention a Peltier device 50 is interposed between the reference body 41 and cell 10 and is energised from a direct current source 51 to reduce the temperature of cell 10 by the required amount with respect to reference body 41. The current supplied to Peltier device 50, which is a known, commercially available unit, is controlled by appropriate means 52, illustrated as comprising a switch 53 and a variable resistor 54. It is apparent that more complex control means may readily be provided if required, for example, automatic control

means to provide a required rate of temperature decrease and/or increase may in known manner respond to temperature-denotive signals developed by a sensor applied to or incorporated in cell 10, possibly in substitution for thermometer 14. It is an advantage of the apparatus described that the Peltier device is conveniently operated at a low direct voltage, for example 6V or 12V, so that control by known semiconductor switching devices is very simple.

The whole of the cooling means 40, 50, together with cell 10, is enclosed by thermal insulation 60 provided by an envelope of thermal insulating material, conveniently expanded polystyrene, to reduce ingress of heat from the ambience. Plugs 20, 21 of cell 10 are made of such a length that they extend to the outer surface of thermal insulation 60, so that illumination and viewing of the interior of well 12 is assisted.

In the application of the apparatus described with reference to Figure 4 to the determination of the freezing point of a liquid, for example an aviation fuel, a sample of the liquid, which may have a volume of only 3 millilitres, is introduced into the well 12 of the cell and, on the assumption that reference body 41 is already at its working tempearture of −15°C, Peltier device 50 is energised to the application of direct current from source 51 so as to reduce the temperature of the block 11 and the liquid contained in well 12, to a temperature below its freezing point, as evidenced by the readily observed appearance of wax crystals within the cell. When freezing is seen to have occurred the cooling effected by Peltier device 50 is reduced, discontinued or even reversed so as to produce an appropriate rate of temperature rise in the liquid. The temperature at which disappearance of the last visible wax crystal occurs is the freezing point of the liquid.

It is found in practice that measurement of the kind described may be made within 15 minutes, whereas the time taken by the present method for determination of the freezing point of aviation fuels, as laid down in the joint specification IP16-ASTM D2386 of the Institute of Petroleum and the American Society for the testing of materials, requires many times as long.

When the measurement has been effected the samples in cell 10 may be replaced by a new sample, for example by flushing out the existing sample by a flushing liquid introduced through passage 15, thus expelling the sample through passage 17. A new sample may then be introduced into the cell through passage 15.

In an alternative mode of operation the samples may be introduced into the cell by applying suction to passage 17 and discharged by applying air pressure to that passage.

In a modification of the testing system illustrated by Figure 4 a viewing cell generally similar to cell 10 may be provided externally of thermal insulation 60 and arranged to be filled with a sample of the liquid to be tested, thus providing for visual comparison of the liquid at ambient temperature with the frozen liquid, under similar viewing condntions.

In further modifications of the testing system illustrated in Figures 2 and 3, and as illustrated in Figure 5, photoelectric means are provided for determining the freezing point of the liquid in cell 10. A photosensor 76 responsive to light passing through the cell 10 from a lamp 75 by way of transparent plugs 20, 21 will receive less light in the presence of any wax crystals within the cell. The signal developed by photosensor 76 is amplified by an appropriate amplifier 77 and applied to a display means, for example a pointer instrument 78 as illustrated. The use of polarised light may be found advantageous when photoelectric sensing is employed. A polariser 79 is placed between a light source and the viewed liquid and an analyser 80 between the liquid and the sensor. The response of the sensor will then vary by a greater amount in the presence of wax crystals than when the polariser and analiyser are omitted.

It is also found advantageous to effect stirring of the liquid within the cell. This may advantageously be effected by means of an electromagnetic pulser 70 which, after the cell has been filled is coupled to the conduit 15*c* through which the cell is filled, this coupling is conveniently effected by providing conduit 15*c* with a conical end fitting 15*d* into which a mating plug 69 provided at the end of a flexible conduit 68 extending from pulser 70 is provided. In this case the discharge conduit 17*c* is arranged to be coupled to a pulse-indicator 81 provided with a plug 80 matable with a conical end fitting 17*d* on overflow conduit 17. Alternatively the tube may be formed of transparent material and a portion is arranged to project beyond the thermal insultion 60 so that the meniscus 82 formed by the liquid within the cell is visible. In this manner pumpability tests can be readily achieved i.e. as the sample is cooling down and the viscosity starts to increase, the fluid in the sight gauge which is pulsating in unison with the sample, slows down and the displacement length continues to decrease, finally dropping to zero when the sample viscosity has increased to the point when it will not flow through the discharge tube, or freezing has occurred.

One electromagnet $70a$ used to drive pulser 70 is advantageously driven from a multivibrator 71 fed from a d.c. supply. The pulsing rate may be controlled by a variable resistor 72 and the energization of the magnet may be signalled by a light, emissive diode 73 connected in shunt with the magnet.

The apparatus of the invention may also advantageously be used as an apparatus for determining the wax appearance point of distillate fuels, at present requiring the elaborate and bulky apparatus set out in American National Standard ANSI/ASTM D 3117.

WHAT WE CLAIM IS:—

1. A testing system for performing a test upon a sample of a material at a temperature other than ambient temperature, in which the temperature of a testing apparatus is arranged to be changed with respect to that of a reference body, which is itself arranged to be held at a temperature different from the ambient temperature, by a Peltier effect unit thermally interposed between said reference body and said device.

2. A system in accordance with claim 1 wherein said reference body, said Peltier effect unit and said test device are enclosed within a common mass of thermally insulating material.

3. A system in accordance with claim 2 wherein visual observation of the material under test is provided by transparent rods extending from said test device to the outer surface of said mass of insulating material.

4. A liquid freezing-point testing system in accordance with claim 3 wherein said test device comprises a metal block having therein a well arranged to be filled with said liquid, said transparent plugs and having one end face within said cell and another end face positioned outside said thermal insulation, and a thermal sensor extending into said well so as to be visible through at least one of said plugs.

5. A system in accordance with claim 4 wherein said plugs are optically aligned.

6. A system in accordance with claim 5 and including a light source arranged to illuminate said well by way of one of said plugs, a photo-sensor arranged to receive light from said well by way of the other of said plugs and display means arranged to provide an indication of the light received by said photosensor.

7. A sysem in accordance with claims 4, 5 or 6 and including a means for producing pulsating displacement of the liquid in said well.

8. A system in accordance with claim 7, wherein a part at least of a duct through which excess liquid may escape from the top of said well is exposed to view and is formed of transparent material.

9. A testing system in accordance with claim 1 and substantially as herein described with reference to Figure 1 of the accompanying drawings.

10. A liquid freezing point testing system substantially as described with reference to Figures 2 to 4 and modified as described with reference to Figure 5 of the accompanying drawings.

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Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1980. Published at the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

1575819 COMPLETE SPECIFICATION

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Sheet 1

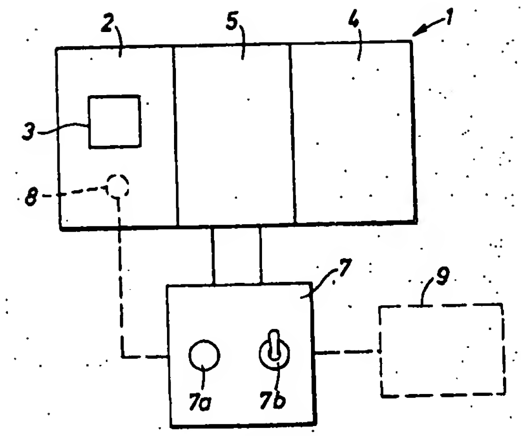

FIG.1.

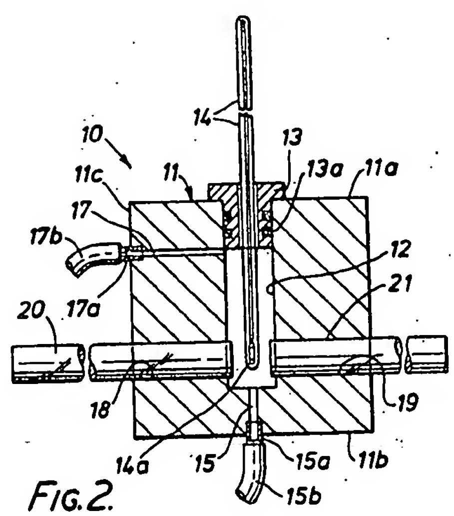

FIG.2.

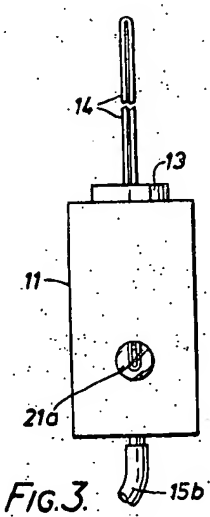

FIG.3.

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Sheet 2

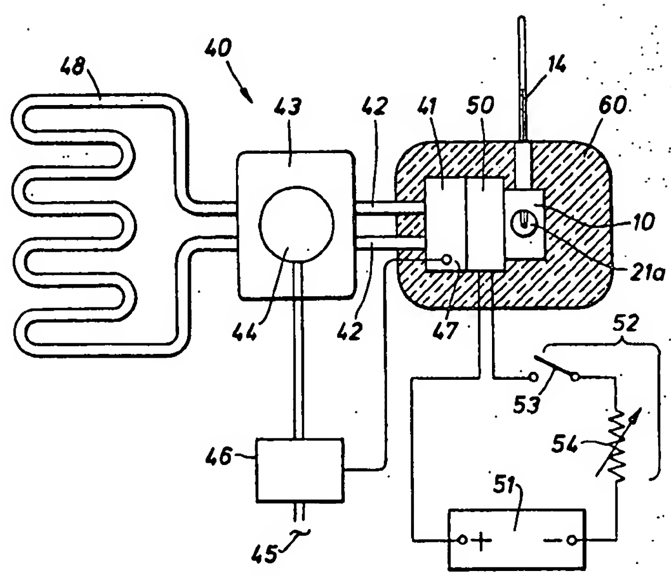

FIG.4.

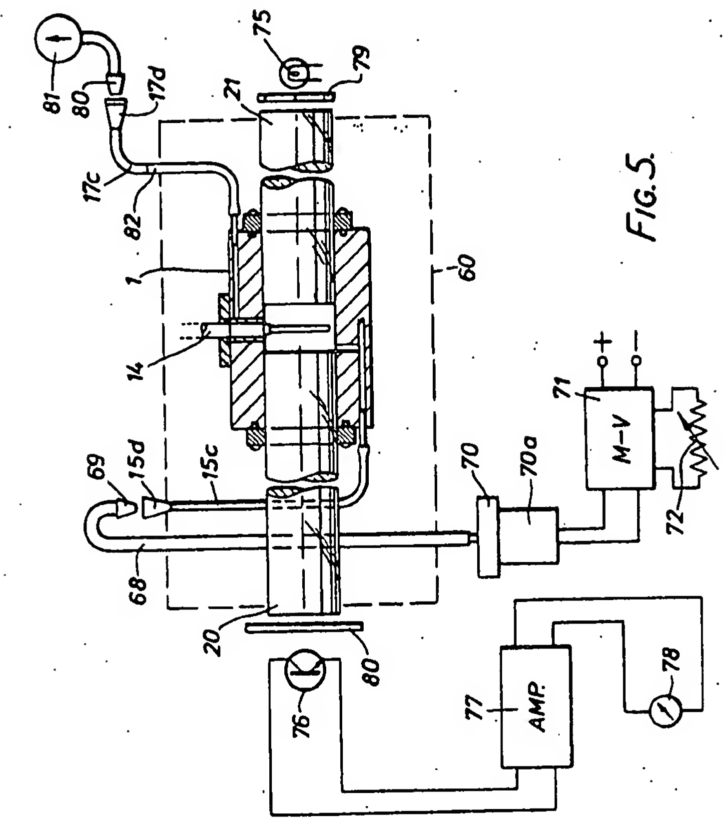
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FIG. 5